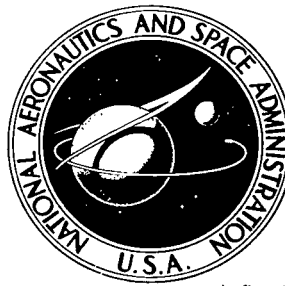


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LOW-ENERGY ELECTRON DIFFRACTION
STUDY OF OXYGEN ADSORPTION ON
MOLYBDENUM (111) SURFACE

by John Ferrante and Gilbert C. Barton

*Lewis Research Center
Cleveland, Ohio*





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ADSORPTION OF MOLYBDENUM (111) SURFACE

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ABSTRACT

Low-Energy Electron Diffraction was used to study oxygen adsorption on, and thermal stability of, the Mo (111) surface. The crystal was subjected to oxygen exposures ranging from 0 to 10^{-4} torr-sec. The oxygen exposures resulted in four new surface structures, a chemisorbed structure and three others identified as chemical faceting into (112) planes. The latter required heating the crystal to 700°C to form completely. Differences from oxygen adsorption on W (111) were observed. Oxidation of the surface at room temperature was demonstrated. The surface was found to be thermally stable when heated for several minutes at 2100°C .

LOW-ENERGY ELECTRON DIFFRACTION STUDY OF OXYGEN ADSORPTION ON MOLYBDENUM (111) SURFACE

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SUMMARY

Low-Energy Electron Diffraction was used to study oxygen adsorption on, and thermal stability of, the molybdenum (111) surface. The crystal was subjected to oxygen exposures ranging from 0 to 10^{-4} torr-second. The oxygen exposures resulted in four new surface structures, a chemisorbed structure and three others identified as chemical faceting into (112) planes. The latter required heating the crystal to 700°C to form completely. Differences from oxygen adsorption on tungsten (111) were observed. Oxidation of the surface at room temperature was demonstrated. The surface was found to be thermally stable when heated for several minutes at 2100°C .

INTRODUCTION

In this study the technique of Low-Energy Electron Diffraction (LEED) was used to examine the thermal stability of, and structural changes upon oxygen adsorption on, the (111) surface of molybdenum. The purpose is to provide information useful in thermionic power conversion, where molybdenum is of interest as a potential electrode material. Oxygen on molybdenum is of interest, since coadsorption with cesium and electronegative gases may be used to provide lower emitter work functions and higher temperature operation (refs. 1 and 2). Another aspect of the experiment is that other LEED studies (refs. 3 and 4) of oxygen adsorption on tungsten and molybdenum have given similar results for the (110) surface; therefore, it would be interesting to see if this behavior would be repeated on the (111) surface. Taylor's study (ref. 5) of oxygen adsorption on tungsten (111) provides a good basis for this comparison. Molybdenum and tungsten have similar physical and chemical properties; therefore, any differences may be of value in understanding surface structural changes.

The stability of a surface is related to its free energy. A higher free energy implies a lower surface stability. Calculations suggest that thermal faceting might occur on the (111) surface since it has the fourth or fifth lowest free energy among the low index plane surfaces in the body-centered cubic structure. Observations were made, therefore, concerning the thermal stability of the (111) surface.

This study also included a determination of the procedures necessary to obtain a "clean" molybdenum (111) surface, an examination of the structural changes with oxygen adsorption as a function of temperature and exposure, and an observation of room temperature oxidation.

EXPERIMENT

Apparatus

The LEED system used in the study was a Varian two grid, fluorescent screen model which is similar in operating principles to the system described in reference 6. The molybdenum crystals used were an ultrahigh purity grade, three-pass refined, with an impurity level of less than twenty parts per million. The crystals were oriented to within 1° of the [111] direction, mechanically and electrolytically polished, and then mechanically fastened to 0.030 inch (0.075 cm) diameter molybdenum supports. The dimensions of the crystals were 0.9 by 0.125 by 0.006 inch (2.3 by 0.320 by 0.015 cm).

Temperatures quoted in the report were obtained with a brightness optical pyrometer that was calibrated with a National Bureau of Standards source. Temperatures below the pyrometer range of approximately 700°C were based on an extrapolation using a correlation between heater current and temperatures obtained with the pyrometer. Temperatures quoted contain a 50°C window correction and an assumed emissivity of 0.37. The estimated accuracy of the measurement is $\pm 25^\circ\text{C}$. A high, direct current power supply was used to heat the sample.

Gases used in the adsorption studies were research grade with an impurity level of less than 100 parts per million. Partial pressures were verified with an Electronics Associates, Incorporated quadrupole mass spectrometer.

Cleaning the Crystal

The crystal and supports were initially outgassed after which the vacuum system could be maintained at pressures in the low 10^{-10} torr range for approximately 30 seconds when the crystal was heated to 1930°C .

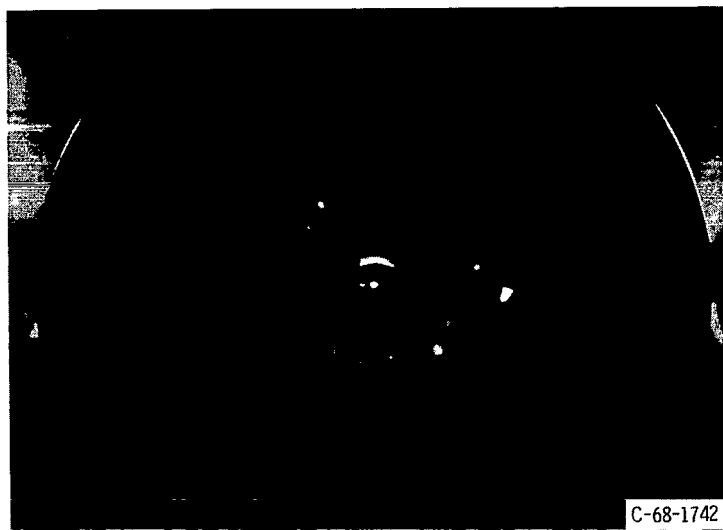


Figure 1. - Contaminant pattern. Beam voltage, 105 volts.

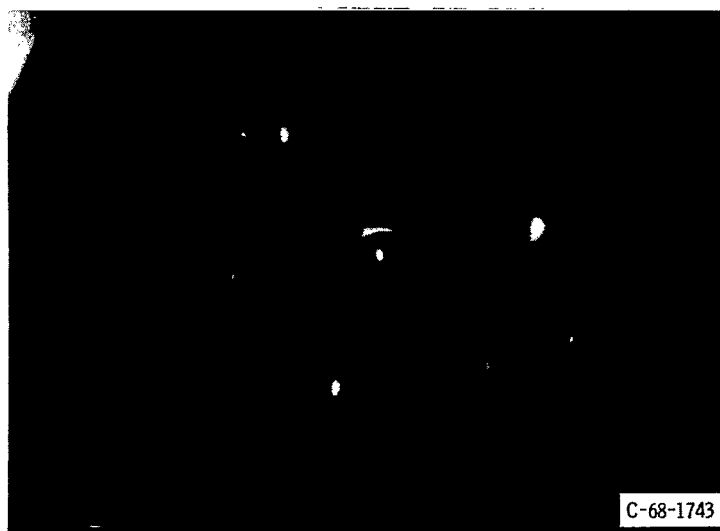


Figure 2. - Pattern for clean structure (C-structure). Beam voltage, 43 volts.

After outgassing the crystal, the diffraction pattern shown in figure 1 was observed. This pattern was attributed to "carbon" contamination on the surface. In order to remove the contaminant, the crystal was then heated in oxygen at a pressure of 1×10^{-7} torr and at a temperature of 1800° C. During this cleaning procedure, a decrease in the carbon monoxide peak was observed on the mass spectrometer. The process removed the "carbon" pattern. However, it reappeared with prolonged high-temperature heating of the crystal in high vacuum. The heating in oxygen was repeated until the "carbon" pattern no longer reappeared with prolonged vacuum heating. After this treatment, the background intensity was quite low; no fractional order beams were observed; and the (111) diffraction pattern reached a maximum in intensity. Thus, it was assumed that the surface was "clean." The clean molybdenum (111) pattern is shown in figure 2.

Procedure for Obtaining Oxygen Adsorption Patterns

In performing the adsorption studies, the molybdenum crystal was exposed to oxygen at pressures from 5×10^{-10} to 1×10^{-5} torr for exposures ranging from 0.25 to 100 Langmuir (0.25×10^{-6} to 1×10^{-4} torr-sec) where 1 Langmuir = 1×10^{-6} torr-second. The fact that oxygen was the primary constituent during the exposure was verified by use of the mass spectrometer. The ionization gage was turned off during and after exposures in order to minimize carbon monoxide contamination. During these periods, the pressure was monitored by the ion pump current.

Following oxygen exposure, it was necessary to heat the crystal to 700° C before a completely ordered structure would appear.

The temperature at which a transition from one surface structure to another occurred was determined by initially subjecting the crystal to an oxygen exposure and then gradually heating to successively higher temperatures until a lower oxygen exposure surface structure appeared.

No desorption studies were performed since the oxygen did not desorb with a pressure burst upon flashing the crystal.

RESULTS AND DISCUSSION

Results of the Oxygen Exposures

Exposing the clean molybdenum (111) surface at room temperature to oxygen caused an increase in the background intensity of the diffraction patterns without, generally, the

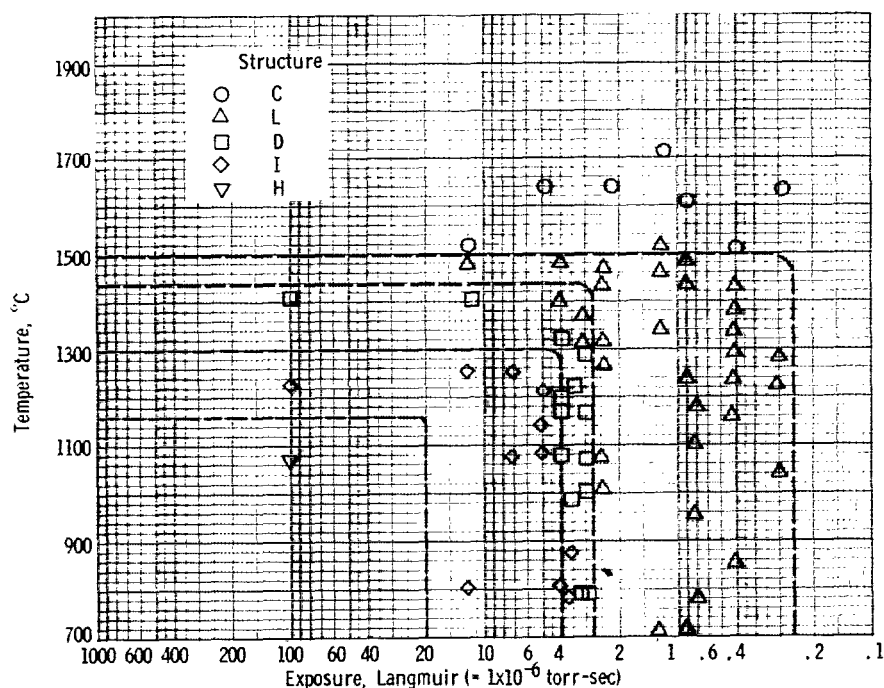


Figure 3. - Existence of oxygen structures as a function of temperature and coverage.

appearance of any new spots. New diffraction spots did appear in the patterns, however, after the crystal was heated slightly. The exception to this behavior occurred at low oxygen exposures for which new diffraction spots could be obtained at room temperature. Nevertheless, even in this case the spots were made much more distinct by heating the crystal slightly.

The appearance of the new diffraction spots revealed four distinct oxygen surface structures with diffuse intermediate ones occurring between them. The different structures occurring as a function of oxygen exposure and temperature are summarized in the graph of figure 3. The procedure used to obtain the data in figure 3 is discussed in the section Procedure for Obtaining Oxygen Adsorption Patterns. The structures are labeled C, L, D, I, and H for the clean molybdenum (111) surface, a low-oxygen-exposure structure, a double-spot structure, an intermediate-oxygen-exposure structure, and a high-oxygen-exposure structure, respectively.

The temperatures at which structure transitions occur, shown in figure 3, are to be taken semiquantitatively, since the transitions are time dependent as well as temperature dependent. It is possible that for longer heating periods the same structural changes might have occurred at lower temperatures; therefore, the transition temperatures shown are to be taken as the points where the structural changes take place readily. The time required to achieve temperature equilibrium varies inversely with tempera-

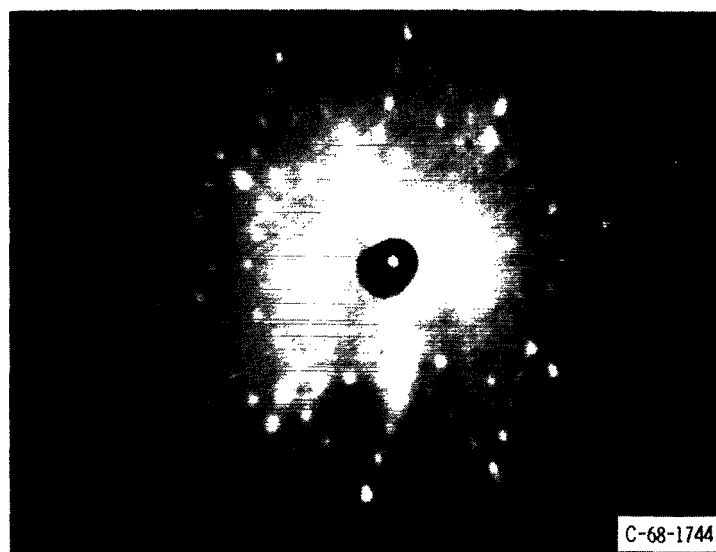


Figure 4. - Pattern for low coverage structure (L-structure). Beam voltage, 73 volts; (00)-spot to right of electron gun.

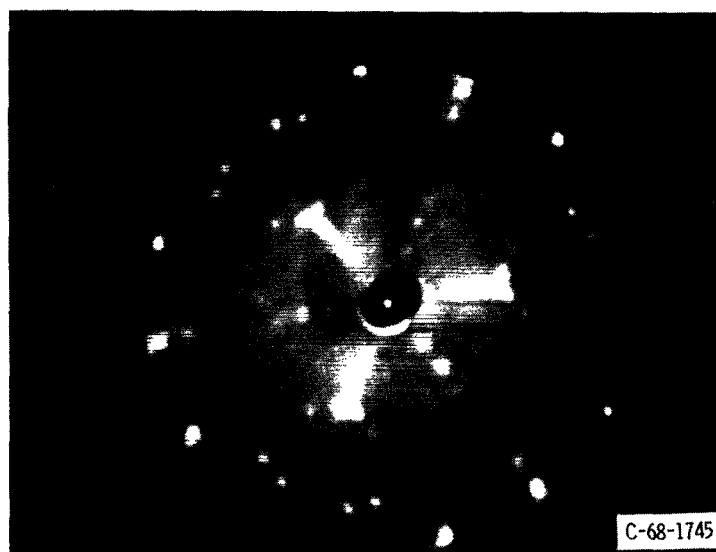


Figure 5. - Pattern for double-spot structure (D-structure). Beam voltage, 94 volts.

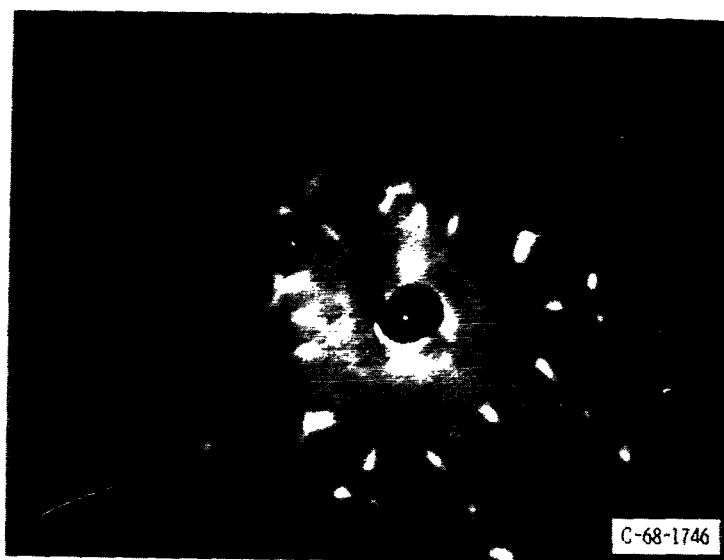


Figure 6. - Pattern for intermediate-exposure structure (I-structure). Beam voltage, 180 volts.



Figure 7. - Pattern for high coverage structure (H-structure). Beam voltage, 127 volts.

ture; therefore, 2- to 5-minute heating periods were used in order to attain temperature equilibrium and minimize carbon monoxide contamination.

LEED patterns of the oxygen structures obtained are shown in figures 4 to 7. The low-oxygen-exposure structure is nonfaceted, whereas the diffraction patterns from the higher-oxygen-exposure structures originate from (112) faceted surfaces as will be shown.

Low-Oxygen-Exposure Structure

The diffraction pattern for the L-structure is shown in figure 4. It was obtained for oxygen exposures between 0.4 and 1.1 Langmuir (0.4×10^{-6} to 1.1×10^{-6} torr-sec) starting from the clean surface. Heating slightly helped to develop a distinct pattern. At exposures from approximately 0.1 to 0.4 Langmuir (0.1×10^{-6} to 0.4×10^{-6} torr-sec), a pattern which is slightly more diffuse than the C-pattern is obtained.

Interpretation of figure 4 is possible by assuming the pattern to be a fourth order pattern produced by the surface being arranged in patches such that the spacing between rows is four times that for the clean molybdenum (111) surface. Another possible interpretation is based on the multiple scattering concept (refs. 7 to 9). In this interpretation, a sixfold symmetry oxygen array, which has a reciprocal lattice vector three-fourths that of the (111) substrate, and the substrate reciprocal lattice are superimposed.

A selection between interpretations based on coverage is not readily available without an accurate knowledge of oxygen sticking coefficients. The fact that some heating is needed to establish a distinct pattern obscures the determination of an optimum exposure for the structure based on a spot intensity measurement.

Oxygen Faceting and the Double-Spot Pattern

At oxygen exposures between 2.5 and 3.6 Langmuir (2.5×10^{-6} and 3.6×10^{-6} torr-sec) and a heating to 700°C , a double-spot pattern shown in figure 5 was obtained.

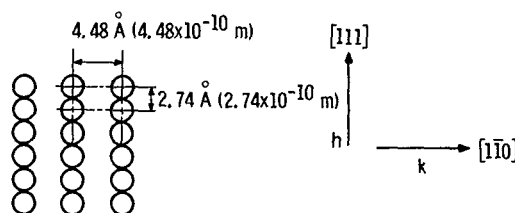
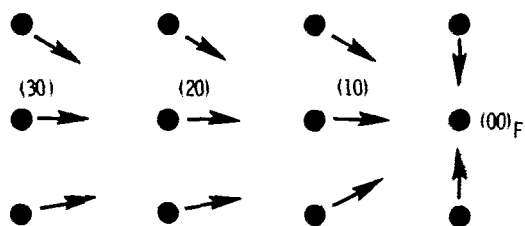
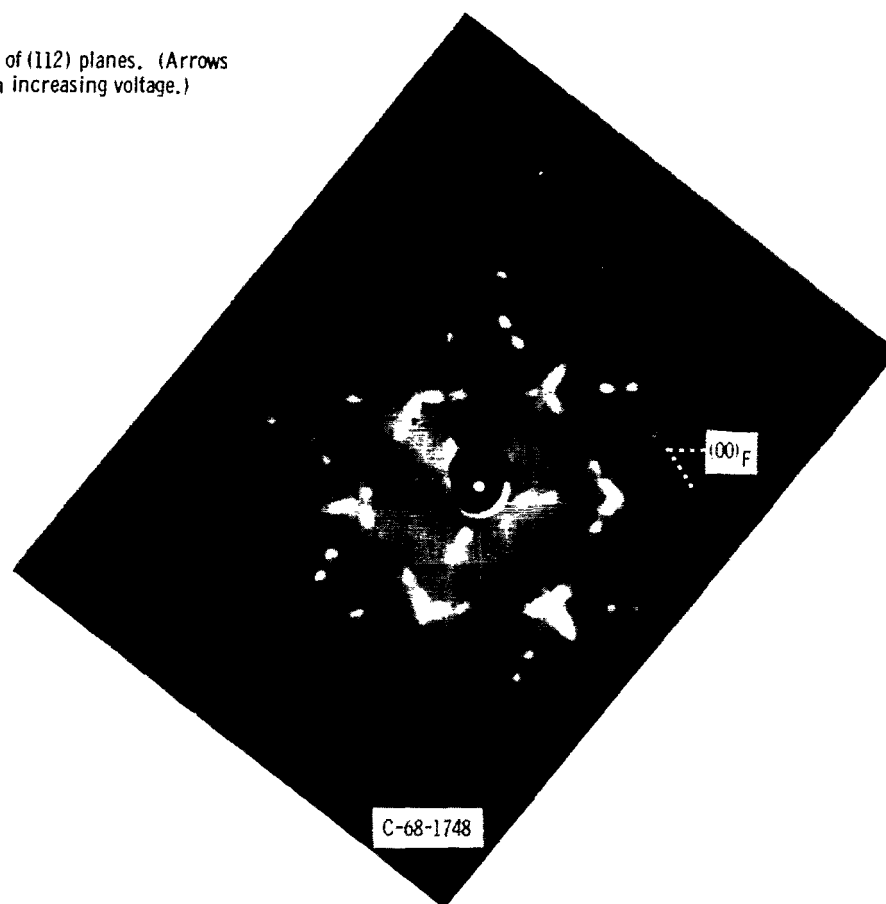


Figure 8. - Arrangement of atoms in $(11\bar{2})$ plane in direct lattice; h and k are Miller indices for surface mesh in reciprocal lattice.



(a) Positions of diffraction spots from one of (112) planes. (Arrows indicate direction of motion of spots with increasing voltage.)



(b) Superposition of three patterns oriented 120° with one another in double-spot pattern taken at 71 volts.

Figure 9. - Comparison between reciprocal lattice for (112) planes and double-spot pattern (D-pattern).

Between oxygen exposures of 1.1 and 2.5 Langmuir (1.1×10^{-6} and 3.2×10^{-6} torr-sec), a pattern intermediate between the L- and the D-patterns is obtained; however, the details of the L-pattern are more easily discerned.

Close analysis of the diffraction pattern in figure 5 shows that it has threefold symmetry and is composed of the patterns from three (112) planes oriented at 120° from one another which occurred as a result of chemical faceting of the (111) surface.

A diagram of the (112) surface is shown in figure 8. Positions of the diffraction spots in the reciprocal lattice for a (112) plane are shown in figure 9(a). The direction of motion of the diffraction spots with increasing beam voltage is indicated. The (00)F spot for the pattern is indicated in figure 9(b). The pattern in figure 9(b) has the correct structure corresponding to (112) planes but is complicated by having the single spots in figure 9(a) replaced by double spots.

The fact that the molybdenum (111) surface has completely faceted into (112) planes was verified by plotting the variation of colatitude angle of the (10), (20), and (30) diffraction spots (fig. 9(a)) as a function of incident beam energy in figure 10; comparing this to the theoretical plot for the (112) surface (ref. 10); and observing that none of the remaining beams follows the molybdenum (111) surface grating equation.

Chemical faceting of the molybdenum (111) surface by oxygen into (112) planes is similar to the result found for tungsten (111) (ref. 5). A satisfactory model of the surface to explain the double-spot pattern has not yet been obtained. It should be noted that all of the oxygen structures observed exhibited complete reversibility with regard to exposures and their reappearance in reverse order after a subsequent desorption. In addition, the results were repeatable with other crystals.

Intermediate-Oxygen-Exposure Structure

This structure is obtained by exposing the (111) surface to form 5 to 20 Langmuir (5×10^{-6} to 20×10^{-6} torr-sec) of oxygen and heating to 700° C. This structure is characterized by a lack of order, that is, the diffraction spots are generally streaked (refs. 6 and 11). An example of the diffraction pattern obtained for this case is shown in figure 6.

This pattern maintains the threefold symmetry of the D-pattern and is again a (112) faceting pattern. The pattern has a (1 \times 2) structure with the double spacing in the less densely packed $[\bar{1}\bar{1}0]$ direction (fig. 8) and normal spacing in the perpendicular $[1\bar{1}\bar{1}]$ direction. For a complete discussion of this nomenclature, see reference 12.

Since the I-pattern is completely a (112) faceted pattern and some lack of order exists, it is difficult to further interpret the structural arrangement of the surface from this pattern. This phase persists for oxygen exposures up to the 20 Langmuir (20×10^{-6} torr-sec) range. Heating to 1300° C causes a transition to the D-phase.

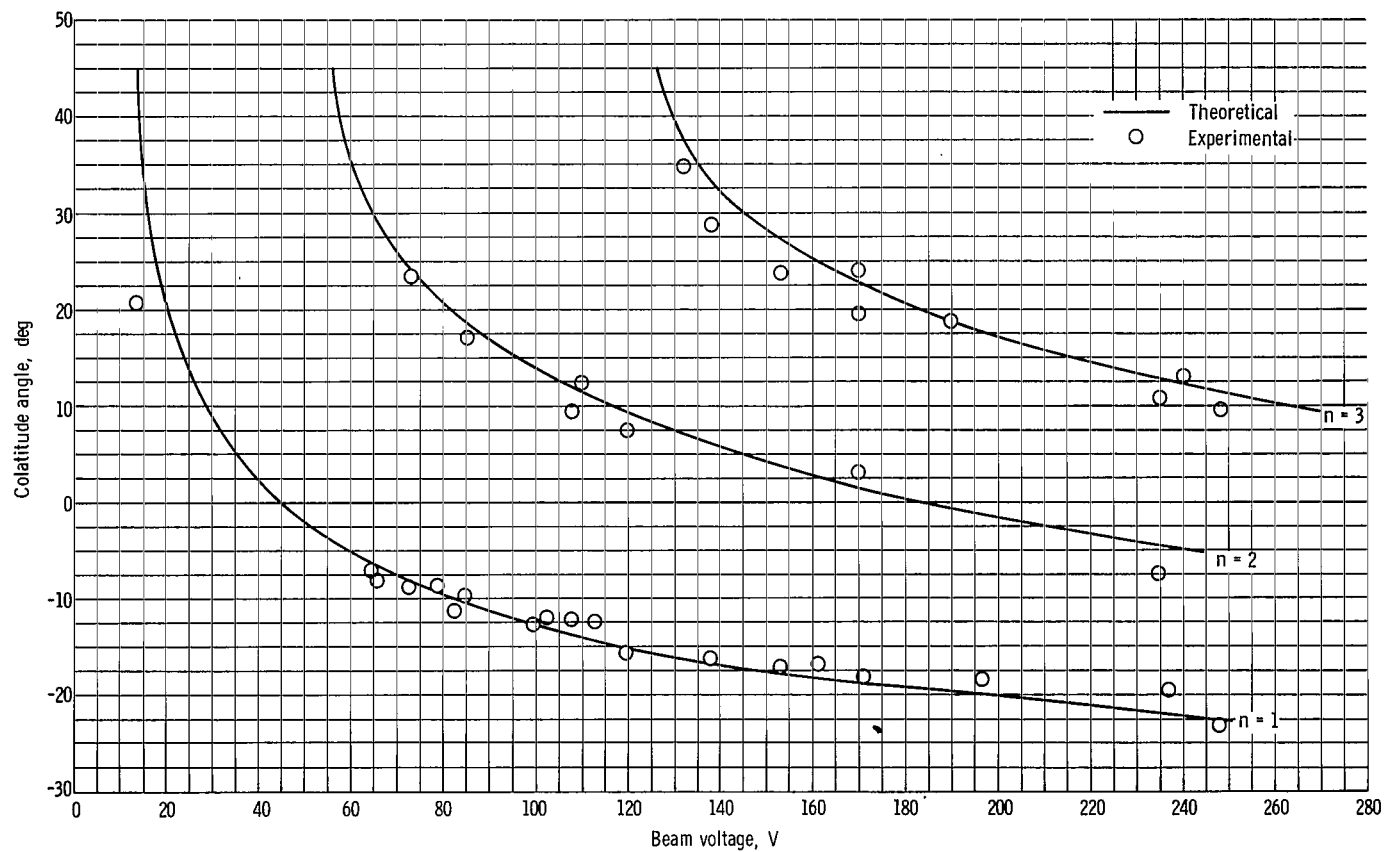


Figure 10. - Colatitude angle variation against beam voltage of various orders for (112) surface with normal incidence in $[111]$ direction.

High-Oxygen-Exposure Structure

Finally, a high-oxygen-exposure structure is obtained with an oxygen exposure of 100 Langmuir (100×10^{-6} torr-sec) and heating to 700°C . The diffraction pattern obtained for these conditions again has threefold symmetry and results from (112) faceting. The pattern, shown in figure 7, can be represented by a (3×3) structure. It has a threefold spacing in the $[\bar{1}\bar{1}0]$ direction and a threefold spacing in the $[11\bar{1}]$ direction. It transforms into the I-pattern upon heating to 1160°C .

To further interpret the structural arrangement of the surface from the H-pattern is likewise difficult because of its streaked diffuse nature.

In an attempt to visually observe the faceting on the (111) surface, an electron micrograph at a magnification of 40 000 was taken (fig. 11). The crystal was given a 100 Langmuir (100×10^{-6} torr-sec) exposure and heated to 700°C , producing an H-pattern. Then it was removed from the LEED system for the purpose of making an electron micrograph. As a control, another crystal whose micrograph is not shown was

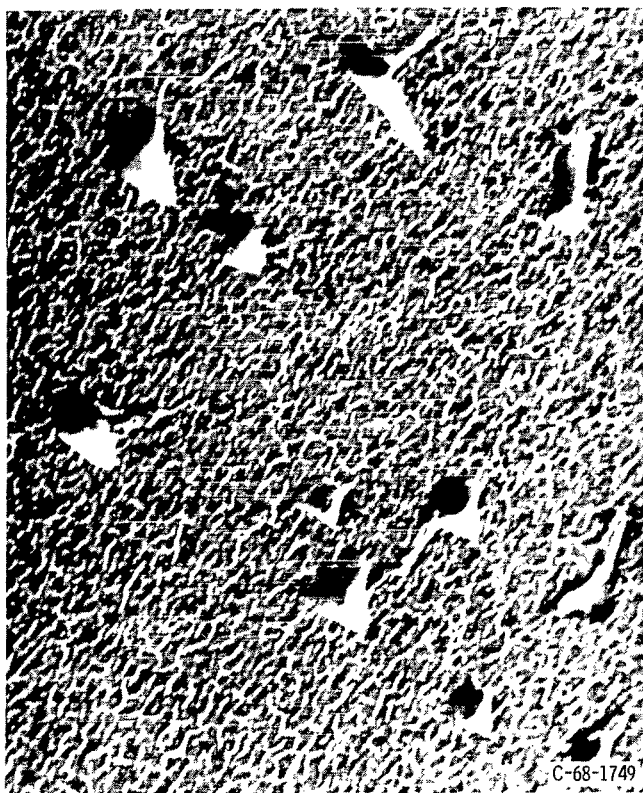


Figure 11. - Electron micrograph of molybdenum (111) surface after 100 Langmuir (100×10^{-6} torr-sec) exposure and heating to 700°C . Magnification, 40 000x.

given the surface cleaning procedure previously mentioned, and a micrograph was made with a C-pattern as a basis.

Figure 1 displays a roughened surface indicating that the faceting has occurred; however, the threefold symmetry appearing in LEED patterns of the H-structure are not observable in the micrograph. The C-pattern micrograph which does not appear exhibited a relatively smooth surface at this magnification.

Oxidation at Room Temperature

A question that might be answered by LEED is whether oxidation occurs on molybdenum at room temperature. This is related to a question of current interest, namely, the oxidation of tungsten at room temperature (ref. 13 and 14).

After room temperature oxygen exposures in the 5×10^{-6} to 100×10^{-6} torr-sec), it was noted that some diffuse spots other than those expected from the molybdenum (111) surface appeared. As a result of these observations, the nature of these room temperature structural changes was investigated in order to determine whether they represented faceting and thus oxidation.

The crystal was exposed to 5 Langmuir (5×10^{-6} torr-sec) of oxygen while at room temperature, and a diffraction pattern was taken. Figure 12 shows the pattern at 72 volts. Note the diffuse spots around the periphery of the pattern. The diffraction beams from the (111) surface are also prominent, that is, the bright triad around the electron gun. Heating the crystal to approximately 425°C caused the spots to become

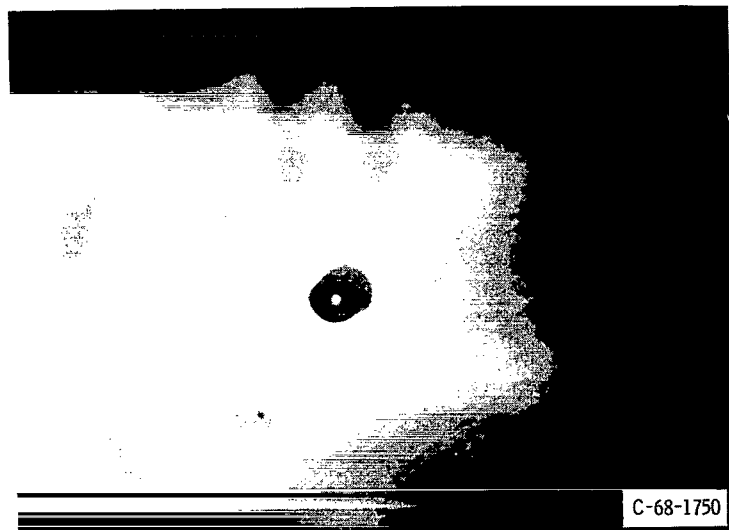


Figure 12. - Room temperature pattern after 5 Langmuir (5×10^{-6} torr-sec) exposure. Beam voltage, 72 volts.

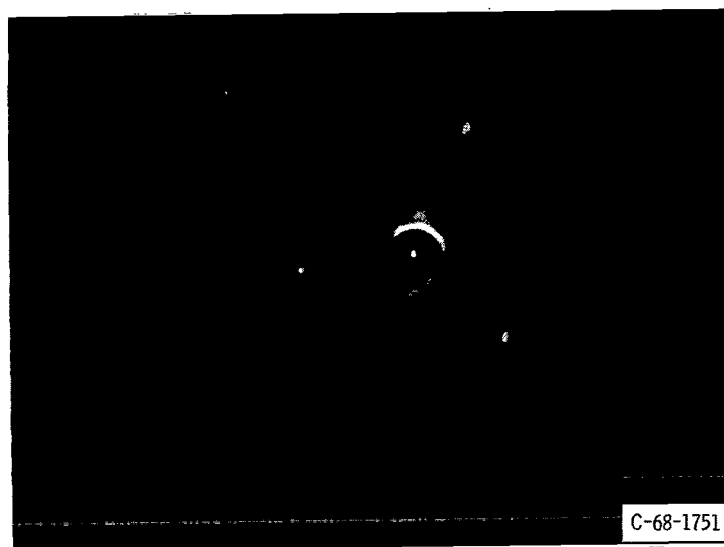


Figure 13. - Pattern after gentle heating at 425° C and 5 Langmuir (5×10^{-6} torr-sec) exposure. Beam voltage, 72 volts.

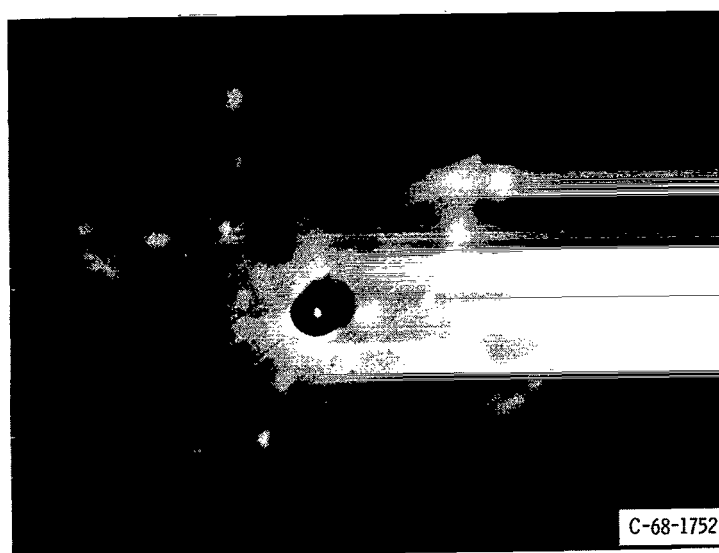


Figure 14. - Pattern after heating to glowing (700° C) and 5 Langmuir (5×10^{-6} torr-sec) exposure. Beam voltage, 72 volts.

better defined (fig. 13). By varying the beam voltage after the heating, it was easy to see that the spots originated from (112) planes. The (111) surface still remained prominent, however. Further heating of the crystal to 700° C completed the (112) faceting (fig. 14), and no diffracted beams from the (111) surface could then be observed. Similar results were obtained with oxygen exposures of 10 and 100 Langmuir (10×10^{-6} to 100×10^{-6} torr-sec).

Tungsten oxides on a tungsten surface were found to have lower desorption energies than oxygen which chemisorbed on the surface (refs. 15 and 16). The same relation is assumed to hold true for molybdenum. The faceted oxygen structures occurring in this study all have lower desorption energies than the unfaceted structure. This is shown in figure 4 by the order in temperature at which structural changes occur. Thus, the faceted oxygen structures are assumed to represent oxides while the L-structure is formed by chemisorbed oxygen. Therefore, the structural changes which have been shown to originate from (112) planes seem to represent some degree of room temperature oxidation for the molybdenum (111) surface.

Thermal Faceting

The (111) surface is the fourth or fifth lowest free energy surface in the body-centered cubic structure (ref. 17). Since surface stability is related to surface free energy, it is possible that prolonged heating at elevated temperatures might cause thermal faceting.

Taylor (ref. 5) observed no thermal faceting on tungsten (111). A similar experiment with molybdenum is of interest since its melting point is approximately 750° C lower than that of tungsten. For comparable conditions, therefore, faceting should take place more easily. Nevertheless, heating for several minutes at 2100° C, a number of times, produced no thermal faceting.

Discussion of Oxygen Structural Changes

As stated earlier, Taylor studied the structural changes resulting from oxygen adsorption on tungsten (111). The major similarities between Taylor's results and those presented in this report are that the surface faceted into (112) planes, that the high exposure patterns are diffuse and streaked, and that it was necessary to heat the crystal to 700° C in order for the facets to form completely. The major differences are that Taylor observed no low exposure nonfaceted pattern, and that his low-exposure faceted structure gives a single-spot pattern. A comparison between the two sets of results for similar exposure ranges is summarized in table I.

TABLE I. - A COMPARISON OF OXYGEN ADSORPTION STRUCTURES BETWEEN
TUNGSTEN (111) AND MOLYBDENUM (111) (REF. 5)

Exposure		Tungsten (111)		Molybdenum (111)	
Langmuir ^a	torr-sec	State of surface	Surface structure	State of surface	Surface structure
0 to 0.4	0×10^{-6} to 0.4×10^{-6}	Nonfaceted	Substrate structure	Nonfaceted	Substrate structure
0.4 to 1.1	0.4 to 1.1	Partially faceted into (112) planes	Overlap of (112) faceting (1x1) structure and substrate patterns	Nonfaceted	(111) fourth order pattern
1.1 to 2	1.1 to 2	Partially faceted	Overlap of (112) faceting (1x1) structure and substrate patterns	Partially faceted	Overlap of L- and D-patterns
2 to 4	2 to 4	Totally faceted into (112) planes	(1x1) structure on (112) planes	Totally faceted into (112) planes	(1x1) structure with double spots
4 to 5	4 to 5	↓	Overlap of (1x1) and (2x3) structures	↓	Overlap of D- and I-patterns
5 to 8	5 to 8		Overlap of (1x1) and (2x3) structures		(1x2) structure I-pattern
8 to 20	8 to 20		(2x3) structure		(1x2) structure I-pattern
20 to 100	20 to 100		Overlap of (2x3) and (1x3) structures		(3x3) structure H-pattern
>100	>100	↓	(1x3) structure	-----	-----

^a1 Langmuir = 1×10^{-6} torr-sec.

The preceding comparisons show that, in spite of the similarities between the materials, molybdenum and tungsten give different results. That this should occur is not completely surprising, since differences in oxygen adsorption characteristics between molybdenum and tungsten have been observed by Morgan and Coomes (ref. 18) using field emission techniques. In addition, the various oxides of molybdenum and tungsten have different crystal structures (ref. 19). For example MoO_3 is orthorhombic, whereas WO_3 is triclinic. The dioxides of molybdenum and tungsten are the only oxides that show the same structure.

A recent report by Chang and Germer (ref. 20) may shed some light on the structures of (112) faceted surface. In their study of oxygen adsorption on tungsten (112), their clean (1x1) structure changes to a (2x1) for a half a monolayer coverage of oxygen. For oxygen exposures greater than half a monolayer coverage, the (2x1) structure goes back to a (1x1) structure, for approximately 1.0 Langmuir (1×10^{-6} torr-sec) exposure, then converts to a (1xn) structures with $n \geq 3$ for higher exposures. The (1xn) could correspond to the (1x2) and (3x3) patterns of this report and similarly to Taylor's higher exposure patterns. The triple spacing in the (3x3) pattern of this report is difficult to see clearly due to the diffuseness and streaking and could indeed be the same as Chang and Germer's (1x3) with some superpositions of the other two patterns of the

threefold symmetry causing the uncertainty. Proper analysis of the double-spot and higher-oxygen-exposure patterns from the (112) faceted molybdenum (111) surface may come from oxygen adsorption studies on the unfaceted molybdenum (112) surface.

CONCLUDING REMARKS

In this study of the oxygen adsorption properties and thermal stability of the molybdenum (111) surface, a number of new observations have been made.

Oxygen adsorption on the molybdenum (111) surface produces four structural changes in the 0 to 100 Langmuir (100×10^{-6} torr-sec) exposure range. The four structures are a low-oxygen-exposure chemisorbed phase and three higher exposure phases which display chemical faceting into (112) planes. It was necessary to heat the crystal to 700°C after an oxygen exposure to produce faceting. The oxygen structures observed were found to be completely reversible in the sense that the structures developing from increasing oxygen exposures could be obtained in reverse order by heating the crystal and causing desorption. On the other hand, heating the crystal in the absence of oxygen at 2100°C for several minutes produced no thermal faceting.

Two observations made that may prove to be useful in understanding surface structural changes are that molybdenum (111) and tungsten (111) give different results in spite of similarities between the materials and that the molybdenum (111) surface exhibited some oxidation at room temperature.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, May 14, 1968,
120-33-02-02-22.

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